THE ANALYST.

MARCH, 1902.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ANNUAL DINNER OF THE SOCIETY OF PUBLIC ANALYSTS.

The annual dinner of the Society took place on Wednesday evening, February 12, at the Hotel Cecil. The President, Dr. J. Augustus Voelcker, M.A., B.Sc., presided. About sixty members and guests were present, among the latter being Sir Ernest Clarke, M.A., Secretary of the Royal Agricultural Society of England; Mr. T. H. Elliot, C.B., Secretary of the Board of Agriculture; Mr. John Lithiby, Assistant Secretary of the Local Government Board; Dr. T. E. Thorpe, C.B., F.R.S., Principal of the Government Laboratory; Mr. W. R. Bousfield, K.C., M.P.; Dr. Henry E. Armstrong, F.R.S.; Dr. J. H. Gladstone, F.R.S.; Professor William A. Tilden, D.Sc., F.R.S.; Professor J. Millar Thomson, LL.D., F.R.S., President of the Institute of Chemistry; Mr. G. T. W. Newsholme, President of the Pharmaceutical Society; Mr. Charles Hawksley, President of the Institution of Civil Engineers; Dr. Rudolph Messel; Dr. Squire Sprigge; Dr. Arthur F. Voelcker; Dr. J. C. Philip; Mr. Thos. E. Holmes, M.B.; and Mr. Aubrey W. Rake. The Right Hon. R. W. Hanbury, M.P., President of the Board of Agriculture, was prevented by indisposition from being present.

The following toasts were proposed: "His Majesty the King," and "Her Majesty Queen Alexandra, their Royal Highnesses the Prince and Princess of Wales and other Members of the Royal Family," by the President; "The Houses of Parliament," by Mr. W. W. Fisher, M.A., responded to by Mr. W. R. Bousfield, K.C., M.P.; "Government Departments," by Mr. Otto Hehner, responded to by Mr. T. H. Elliott, C.B., and by Dr. T. E. Thorpe, C.B., F.R.S.; "The Society of Public Analysts," by Professor William A. Tilden, D.Sc., F.R.S., responded to by the President; and "The Guests," by Mr. Bertram Blount, responded to by Sir Ernest Clarke, M.A., and by Dr. Henry E. Armstrong, F.R.S.

THE PRESIDENT'S ANNUAL ADDRESS.

(*Delivered at the Annual Meeting, January* 22, 1902.)

In following, as I do, in the presidential chair those, my predecessors, who were, one and all, closely connected with the work and the relations of the Public Analyst in the official and limited sense of the term, I cannot fail to have noticed, associated with my selection for the office, features both of advantage and disadvantage. In the first place, I can regard my appointment to this position of honour as a mark of the growing expansion of this Society, of the widening of its bounds, the enlargement of its interests, and the nearer realization of the first of its objects as defined in its constitution. The first object of the Society is therein stated to be "to cultivate the study of analytical chemistry, by the holding of periodical meetings, and by the publication of a journal devoted mainly to analytical chemistry." It was well, indeed, and, in fact, almost necessary, that the class who should first band themselves together, for their common interests, should be those whose official positions brought them into relation with one another and the public, and hence it is that the earlier days of the Society were more essentially those of the Public Analyst in the restricted sense, and that the papers read and the discussions held were mainly those concerned with the official work of the then newly-created and, I am bound to say, somewhat imperfect and frequently-abused individual—the Public Analyst. But, as time has gone on, so have individuals improved and gained by experience, so have methods improved and new ones been devised, and the Public Analyst of to-day is no longer the Public Analyst of twenty, or even ten, years ago. More than this, it has been realized, both within this Society and to an increasing extent outside it, that a man to be a Public Analyst in the true sense must be not only something more than a mere machine, capable of evolving certain analytical results by stereotyped methods, but that he requires to be in every sense a chemist by training, and to have this enforced by subsequent practical experience; that he is a thinking and reasonable being; and that he is an impartial and valuable servant appointed in the public interest. With this change has come about also a change in the attitude of the Public Analyst himself. He finds it necessary that he should be not merely the master of a certain routine of work of limited application, and be able to analyse and talk about the analysis of a limited number of common articles like milk, butter, and spirits, but he finds, too, that he has to become an analyst in the wider sense, and that his real object must be the same as that of the Society—"to cultivate the study of analytical chemistry." Hence it is that anyone now turning over the pages of the official journal of the Society, the Analyst, or looking at the names of the members of the Society, will find a wider sphere now embraced, and that, with all due deference to and appreciation of the valuable work done by our old friend Dr. Vieth, and since ably carried on by his successor, Mr. Richmond, the discussions of the Society have travelled far beyond the perennial topic of "milk." From being a Society, as originally formed, to further the interests and knowledge of a special class, the Society of Public Analysts has felt that its members can no longer have limited views,

but must be analytical chemists in the wider sense, and have interests in common with all those who devote themselves to this branch of chemical science. This has been well reflected in the ANALYST, which is the one journal in this country that is devoted to the advancement of analytical chemistry, and which holds now an accepted and acceptable position among the scientific literature of the day. The papers read before the Society, as the list that follows will tell, and the discussions which have taken place on them, show the same broadening of interest, and I may lastly look upon your selection of myself as your President as an evidence of this. Not myself holding any appointment as Public Analyst under the Sale of Food and Drugs Act, I may yet claim to be a Public Analyst in the wider sense to which you, in your development of the aims and interests of the Society, have given expression, and it was this knowledge that led me cheerfully to accept the honour you wished to put upon me.

But while there may have been, thus far, an advantage to the Society, I feel that there has been a corresponding disadvantage in the departure that has been made—now for the first time. My not being a Public Analyst has, I fear, thrown me, in some respects, out of touch with your particular needs, and out of full appreciation of the often difficult duties you are called upon to discharge. But let me say that you, at least, have not let this be apparent to me, and I have now passed through my first term of office with a deeper sense than ever I had of the ability, the value, the necessity, and the conscientiousness of the Public Analyst as a class. Not that there is not still much that might be improved, that a higher standard of professional conduct and of consideration one for another might not be cultivated, that there might not be less of self-seeking and tendency to underbid one another, and to forget what is due to the profession, in the desire to affix this or that appointment. But these are matters which cannot be improved by the enforcing of any strenuous regulations, or by entering into any compact, but which must be left to the growth of a better feeling and of a higher sense of honour among professional men, and also largely to the force of example.

It has been, then, my genuine pleasure to preside this past year over your deliberations, to take my part in furthering your endeavours, and to do what I could towards maintaining the dignity of the office held by my distinguished predecessors, and to improving the position of the analytical chemist in the eyes of the public and in his official relations.

As regards the Society itself, there has been no marked accession of members, for only nine fresh names have been added, as against twenty in 1900, while the removals have been seven in number—viz., three by death, two by resignation, and two on account of non-payment of subscription. It is, on the other hand, matter for satisfaction that the new list of members standing for election at present contains six fresh names. By death the Society has lost three members, Mr. Norman Leonard, Mr. E. W. Martin, and Dr. G. Harris Morris. Mr. Leonard had long been known to us, and was a frequent contributor to the pages of the ANALYST. He held the post of Public Analyst for Northampton, and was closely associated with our respected ex-President, Dr. Thomas Stevenson. Mr. Martin was one of our American members, and so less known to us personally; but in Dr. Morris we lose one whose work was

well known, and for whom, especially in more recent days, we felt a special sympathy. In the domain of plant physiology Dr. Morris was for many years associated with Dr. Horace Brown, F.R.S., in the latter's classic researches, and then and subsequently his work in connection with the chemistry of brewing showed his great ability, energy, and power of research.

In addition to nine new ordinary members enrolled, we have had the pleasure of including among our honorary members Dr. T. E. Thorpe, F.R.S., the head of the Government Laboratory. Not alone for the recognition shown us do we welcome Dr. Thorpe, but also because, in his official position, he has already given us proofs of his desire to co-operate with us in our work, and to bring about a better understanding between Public Analysts and Government Departments. We may sincerely look forward to much good resulting, alike as regards ourselves and the public interest, from the more ready interchange of views on analytical methods and practices which has characterized Dr. Thorpe's tenure of his high official post, in the more frequent association of leading members of our Society in public inquiries, and in the holding, from time to time, of conferences to settle debatable points, as was the case with the recent one on the margarine question.

The finances of the Society, thanks largely to our Honorary Treasurer, continue in a satisfactory condition, but it is necessary to remind members that there is need of a larger accession of new members to fill the place of those removed, to bear the considerable expense attaching to the issue of the ANALYST, and to provide for the useful development of its work. The ANALYST has well maintained the position it has gained, and it is now more than ever recognised as the journal to which one would naturally refer on points of new analytical methods or the examination of new and special materials. The work of abstracting from other sources has been kept well up to date, and to the Editor, Dr. Sykes, for his judgment and unremitting attention to make the ANALYST a worthy and useful journal, the chief credit is due. As a member myself, for the first time this past year, of the Editorial Committee, I can speak to the great trouble taken by almost each member of that Committee in revising the proofs, in making suggestions in and revisions of the abstracts, etc., and I have frequently been greatly struck by the accumulation of valuable experience and knowledge brought to bear on the subject-matter of the different issues as they come before the committee.

The list of papers read before the Society at its different meetings during the past year is as follows:

February 5.—"On the Authority of the British Pharmacopœia as a Standard under the Sale of Food and Drugs Act." By Alfred H. Allen.

"The Physical State in which Fat exists in Cream." By H. Droop Richmond and Sylvester O. Richmond.

"On the Composition of Dutch Butter." By John Clark, Ph.D.

"Note on the Proximate Analysis of Cloves." By A. McGill, B.A.

March 6.—"The Determination of Dissolved Oxygen in Water in Presence of Nitrites and Organic Matter." By S. Rideal, D.Sc.

"Some Analyses of Oatmeal." By Bernard Dyer, D.Sc.

"The Detection and Estimation of Preservatives in Milk." By M. Wynter Blyth, B.A., B.Sc.

April 3.—"On the Maumené Test for Oils." By C. A. Mitchell, B.A.

"Some Arsenic Estimations relating to Malt Kilns." By Thomas Fairley.

"On Gutzeit's Test for Arsenic." By F. C. J. Bird.

"The Aëration Test for Effluents." By S. Rideal, D.Sc.

May 1.—"Alkaline Waters from the Chalk." By W. W. Fisher, M.A.

"Citron Oil." By Herbert E. Burgess.

"Note on the Reduction of Iron Salts." By Gilbert T. Morgan, D.Sc.

June 5.—"Arsenic in Coal and Coke." By Alfred C. Chapman.

"Public Analysts' Records." By J. F. Liverseege.

"The Use of Partially Sterilized Milk Cultures in judging the Purity of Water." By H. Droop Richmond.

November 6.—"The Composition of Milk." By H. Droop Richmond.

"The Determination of Carbon in Steel by Direct Combustion." By Bertram Blount.

"*Enteritidis Sporogenes* as Evidence of Sewage Pollution." By M. Dechan.

December 11.—"A Contribution to a Knowledge of the Chemistry of Cider." By Alfred H. Allen.

"On the Mannitic Fermentation of Wine." By Philip Schidrowitz, Ph.D.

"On Cocoa Extracts." By E. G. Clayton.

"Note on Commercial Zinc White and other Pigments." By E. G. Clayton.

A glance at this list will show the varied nature of the subjects dealt with, and, while a large number of them have been, of course, concerned mainly with the work of the Public Analyst in relation to the Food and Drugs Act, yet a considerable number of them have been on subjects of wider application. Such, for instance, were the exhaustive paper by our late President, Mr. Fisher, on Alkaline Waters from the Chalk; that on the Chemistry of Cider by Mr. Allen; two papers by Dr. Rideal on the Determination of Dissolved Oxygen in Waters and on Aëration of Effluents; and, not least important, the interesting paper by Mr. Blount on the Direct Estimation of Carbon in Steel. Nor were matters of special interest to Public Analysts neglected, for we had a useful discussion introduced by a paper from Mr. Allen on the Value of the British Pharmacopœia as a Standard; the important and then burning question of the presence and determination of arsenic in foods and fuels was brought up; and, lastly, Mr. Richmond gave his annual résumé of his work of the year as concerns milk, but supplementing it with many matters of interest which travelled far beyond the merely statistical area.

As regards the meetings themselves, it is with pleasure that an improved and increasing attendance at the ordinary gatherings can be chronicled, and a further satisfactory feature has been the interest evoked in the discussions which have followed the reading of papers, and which has done much to make these latter profitable. The more numerous attendance has been due in no small measure to a happy inspiration of our Editor, Dr. Sykes, who, by the formation of a Dinner Club, which meets after the usual Council meeting is over, and before the evening meeting,

brings the members together in friendly company, and contributes greatly to the promotion of kindly feeling and to better acquaintance of one member with another. The Council would take the opportunity of letting this be better known, and that all members of the Society can, at a small cost to cover postage, become members of the Dinner Club, and will be regularly informed of place and time of meeting.

The Annual Dinner of last year, which had been fixed for February 5, was, owing to the national mourning following on the deeply-lamented death of Queen Victoria, abandoned for the year. It is now fixed for February 12, and I am happy to say that we shall be honoured then by the presence of Mr. Hanbury, the President of the Board of Agriculture, the Secretary of the Board, and other distinguished guests.

And now it is fitting that I should review the principal events of the past year so far as these concern the position of our Society and its members in their public relations.

The only completed act of legislation which has affected us as Public Analysts is the framing of regulations by the Board of Agriculture in respect of milk. But other matters which have been under consideration, and will probably shortly have effect given to them, have seriously concerned us, and to them reference must also be made. These are the report of the Committee on Preservatives and Colouring Matters in Articles of Food, the inquiry into the cases of poisoning at Manchester through arsenic found in brewing materials, the Departmental Committee of the Board of Agriculture on Butter, and the Poisons Committee of the Privy Council.

Taking first the regulations with regard to milk, it will be remembered that a Departmental Committee was appointed early in 1900 by the Board of Agriculture to draw up regulations which should govern—in respect of their quality—the sale of milk and cream. Of this Committee Lord Wenlock was the chairman. Though, somewhat strangely, not numbering among the members a single Public Analyst in the strict sense, the Departmental Committee availed themselves very largely of the evidence and experience of Public Analysts and others belonging to our Society, and, it may be added, gave, in their report, great weight to the views of these representatives and to the conclusions adopted, with general agreement, by the Society for the guidance of its members. Ultimately the report of the Departmental Committee, issued in March, 1901, recommended a governing limit for milk of 12 per cent. of total solids, and that if a milk failed to give that quantity it should be further inquired into; that if then it be found to have less than 3·25 per cent. of fat, or to have less than 8·5 per cent. of non-fatty solids, a presumption should be raised, until the contrary was proved, that it was not genuine. In regard to cream, the Committee did not advise the fixing of any definite limits, but wished to prohibit the artificial thickening of it. Skimmed and separated milk were to have at least 9 per cent. of total solids, and condensed milk not less than 10 per cent. of fat and 25 per cent. of non-fatty solids.

The President of the Board of Agriculture did not see his way to adopt the limits which the Departmental Committee had advised, and finally, in August, 1901, regulations were issued by the Board of Agriculture, requiring milk, in order to be

considered genuine, to contain not less than 3 per cent. of fat and not less than 8·5 per cent. of non-fatty solids. Skimmed and separated milk are in future to contain not less than 9 per cent. of total solids, but no regulations are made applying to cream or condensed milk.

That such result can be wholly satisfactory to those of us who have experience of the working of the Act is more than can be expected, and there must be a general feeling that the limits might very well have been extended, as proposed by the Departmental Committee, and a great deal of adulteration of milk been prevented, while, at most, the fixing of the higher limits would only have raised a "presumption" of want of genuineness, leaving further inquiry to be made. The opportunity, we think, might well have been taken to strike a blow at systematic "toning down" of milk, and to have given the country a higher quality of milk. Still, there is no getting away from the fact that the proposal to set the limits higher frightened, as we believe unnecessarily, the producers of milk—*i.e.*, the farmers—and they were hardly prepared for so marked a change as was proposed, and in deference to their views the Board of Agriculture declined to at once adopt the higher figures. Something, too, has now been gained in a definite pronouncement that milk that is sold shall have at least 3 per cent. of fat. The absence of regulations as to condensed milk is, however, to be regretted, for there can be little doubt that it is in the sale of condensed milk, deprived to a large extent of its nutritive qualities, and with the deficiency made up by the unrestricted addition of sugar, that great injury is done to infant life, especially in the poorer districts of large towns.

Another matter of kindred importance is the inquiry of the Departmental Committee of the Board of Agriculture into the regulations that shall guide the sale of butter. The Society of Public Analysts is represented on this Committee by one of its former Presidents. The less intricate part of the inquiry—that concerning the amount of water allowable in butter—has already been dealt with; but the more complex one—the composition of the fats and their determination—remains; and this, in view of the continual recurrence of what may be called "abnormal" butters, will inevitably present much difficulty. It is very clear that we know comparatively little about butter, and that there is much that has still to be made the subject of patient research and practical inquiry before we can properly formulate any clear distinction between what is butter and what is not. The Society of Public Analysts was asked to express its views through two of its representatives, and Dr. Dyer and Mr. Hehner have tendered their evidence on behalf of the Society.

Yet another Departmental Committee, that on Food Preservatives, appointed by Mr. Chaplin in 1899, has been conducting an inquiry in which analysts are much concerned, and has just reported its recommendations. The evidence of several prominent members of our Society was given before this Committee, and the recommendations arrived at, it may be said generally, meet with the approval of this Society. Special satisfaction will be felt at the recommendation which advocates the entire prohibition of the use of preservatives or colouring matter in milk. Next to this in importance is the suggested prohibition of formaldehyde and the limiting of the amount of boracic preservatives in cream, butter, and margarine. It is

sincerely to be hoped that these recommendations of the Committee will be carried into effect.

The serious outbreak of illness in Manchester and district towards the close of 1900, and which was traced to the presence in beer, through the brewing materials used, of arsenic, caused great concern and anxiety to Public Analysts throughout the country, and for some months, it may be said, the laboratories of many members of our Society were turned into beer depots and arsenic-testing establishments. Needless to say, the work was of a most delicate nature, and on the Analysts' reports hung important issues. As an outcome of the spread of the epidemic, and the serious results that in many cases followed, a Royal Commission was appointed to inquire into the whole matter; and here, again, members of this Society rendered great service by the evidence they tendered, and still more by the immense amount of work which they did in the effort to arrive at definite conclusions as to the occurrence of arsenic in different materials used for brewing, and in other articles of food and drink, and as to the definite quantitative estimation of small amounts of arsenic in such materials. The papers read before this and kindred societies testify to the vast amount of work done by members of the Society to arrive at the truth, and this culminated in the appointment, jointly by the Society of Public Analysts and the Society of Chemical Industry, of a Committee to examine and report upon the best methods for the estimation of arsenic in minute quantities. This Committee has just recently presented its report, which has been brought before both Societies and will shortly be published. Meanwhile the best thanks of this Society are due to those of its members who were on the Committee, and they may be congratulated on the careful and exhaustive nature of their work, and on the unanimous conclusions come to. It has now been announced that the Royal Commission, above alluded to, has appointed a Committee to conduct on their behalf an inquiry into, and formulate methods for, the determination of arsenic in food materials. Among the experts named are Dr. Thorpe and Professor Tilden. I think we cannot do better than refer the Committee to the report of the Joint Committee of the Society of Chemical Industry and Society of Public Analysts.

During the past year several new forms of adulteration have come to the front, new forms of food articles have been introduced, and legal decisions have been given, all of which affect the position of Public Analysts, and to which brief reference may be made. Perhaps the new material to which most attention has been drawn is that known now as "Milk-blended butter," but sold at first simply as "butter." This article is made by taking colonial butter and churning it up with milk, preservatives being added to prevent the milk "souring." The product contains from 20 to 25 per cent. of water, instead of the 16 per cent. which analysts generally agree should be an extreme limit for butter. By a decision of the High Court of Justice, in August, 1901, this material could no longer be sold as "butter," and the term "milk-blended" was thereupon ingeniously introduced to allow of the material being sold, as, if not sold as butter, it could not, by the provisions of the Margarine Act, be offered as margarine, the proportion of butter in it exceeding the allowed 10 per cent. Hence the importance to the sellers. Many have been the proceedings taken against this article, and various have been the decisions given, and the point at issue cannot

really be satisfactorily settled without a High Court decision being given upon it. It cannot, however, but be clear to anyone who has knowledge of the article that the incorporation of milk with butter is done purely with the object of making, as Mr. Justice Wills said, "a spurious profit," and it is equally clear that the qualifying term "milk-blended" is a misleading one, and does not convey to the public the idea of the true nature of the article, or that it is butter which has been reduced in value and composition by the addition of milk and preservatives to it. One may very rightly ask where the mixing is to stop; for if 10 or 20 per cent. of milk may now be added to butter, and the mixture be sold by a name not disclosing its real nature, what is to prevent 30, 40, or even more, per cent. being added if there be found means of incorporating it?

The same point has quite recently been raised with regard to the latest form of adulteration—the addition of water to margarine. If butter may not have more than 16 per cent. of water, why should margarine be allowed to have more? and if it be maintained that there is no limit for water in margarine, where is the addition to stop? Why may not 30 or 40, or more, per cent. of water be put in if 20 per cent. is allowed? The necessity of restriction in this direction is clearly apparent.

The difficulties of the butter question have already been alluded to; but just as the analyst was beginning to think that he was coming to some understanding about butter and the discrimination of butter-fats and animal and vegetable fats and oils, the introduction, in large quantities, of butter from Siberia, made under conditions of climate, temperature, etc., vastly different to those of our own country, has brought in further complications, for this butter has been found to give results, as regards the volatile and non-volatile fatty acids, of quite abnormal nature. The question thus opened up is a very serious one. It is now recognised that butter made under exceptional conditions, as, for instance, in a very cold climate, and when cows are kept out in the open longer than is usual, will give analytical results which do not conform to those of butter as ordinarily produced; and though it may be nothing but the product of the churned cream of the milk of the cow, it may yet approach in composition to margarine or other fats. Inasmuch, therefore, as the analyst has not the means of knowing where his sample comes from, the discrimination of margarine and foreign fats from genuine butter is rendered still more difficult. It remains then, it would seem, a question of public policy whether such "abnormal" butters should be allowed to enter and be sold as butter, or whether, as was done in the case of the Margarine Act, exclusion of certain kinds should not be insisted on and limits be fixed to which all genuine butter should conform. I must not pursue this matter further here, but leave the rest to the consideration of the Departmental Committee on butter regulations, who, however, I anticipate, will find their task no easy one.

The last form of adulteration to which I shall refer is that of glucose in marmalade. After the obtaining of a conviction in the police-court at Worthing, and the upholding of the conviction at the Horsham Quarter Sessions, for the sale of marmalade to which glucose had been added, the same not being declared, the case was taken on appeal to the High Court, King's Bench Division. The Lord Chief Justice, in giving the decision of the Court, quashed the conviction on the ground that glucose was not found to be injurious to health, and that, as the object of the Sale of Food

and Drugs Act was the protection of health, it ought not to be construed as an instrument of oppression. It was allowed that a different article had been supplied to that asked for, but the Justices held that the article was not an inferior, but possibly rather better one, and that there was no evidence of inferior quality or adulteration in the ordinary sense of the word. See 1901 p. 332 –

Though it is not for me to discuss the legal bearings of the case, I am sure all Analysts will agree with me that the decision of the Lord Chief Justice gives a quite different interpretation to the Act to any we have ever associated with it; and if the test to be applied in the case of alleged admixture without declaration of the nature of the admixture is merely whether the material is injurious to health or whether it is superior or inferior to that asked for, there is no telling where admixture will cease. It would be quite easy to show that vegetable marrow and carrots were not injurious to health, and gave, when mixed with fruit, quite as good a jam as whole fruit; but, for all that, the material sold would not be of the "nature, substance, and quality demanded" by the purchaser. This decision, following on the important point gained in the case of golden syrup, whereby glucose, if used, must be declared to be present, seems to me a most unfortunate one so far as the public and the Public Analyst are concerned.

Other legal decisions that bear on the Public Analyst's position concern the sufficiency of the present form of certificate, and the non-obligatory nature of the instruction to insert the weight of the article sent for analysis.

In our relations to Government Departments and to outside bodies several matters of special interest have come to the fore. We cannot help seeing, and with some satisfaction, the tendency to bring our work into closer connection with the Board of Agriculture, in place of the Local Government Board, which latter body discharges duties, as affects the Public Health, more of a medical than a chemical character, and has relation, thus, rather to the Medical Officer of Health than to the Public Analyst. The desirability of separating, as far as possible, the work of the Medical Officer of Health and of the Public Analyst has been more and more apparent, and has formed the subject of strong representations to the Local Government Board both from the Institute of Chemistry and the Society of Public Analysts. Reference to the action taken is made in the ANALYST for December, 1901, and it is confidently believed that the representations made will not be without good effect. 333

One other matter I must briefly touch upon, as it is one that depends for remedy upon our members themselves, and on the growth and cultivation of a higher sense of professional status and its requirements. I refer to the acceptance by members of the Society of appointments under terms which are not worthy of the dignity or responsibilities of the office. So long as there are men who are willing to accept appointments under such conditions, one can hardly blame Corporations and Local Authorities for encouraging them to underbid one another. But this is not "cricket," as one might say, and we might join with Rudyard Kipling in bemoaning the decadence of a true spirit of duty and obligation.

I have endeavoured to show in my address that the work of the Society is going on well and steadily, and that more and more demand is being made upon its members to give the benefit of their experience and opinion on matters of public

inquiry, Departmental Committees, and the like, and I feel sure that with hearty co-operation and the raising of the standards alike of efficiency and honour the analytical chemist and Public Analyst will become an increasingly useful and necessary individual. One word of warning I would interpose. There is just a danger that, with all the fixing of limits towards which legislation seems to tend, there will be a disposition (to which, indeed, expression has been occasionally given) to degrade the Public Analyst's position to that of a mere recorder—a machine, in fact, to turn out certain figures, and to leave these, without comment or interpretation of their significance and bearing, to be interpreted by others unacquainted, necessarily, with the real points at issue. It may be well to have limits defined, but the application of these must be governed by the judgment of those who have had the responsibility of making them. To regard the Public Analyst merely as one who has to supply certain figures and to have no further voice, if his authorities wish to consult him, is to altogether misunderstand his true position, and to surrender the work by which it has been gained, and I sincerely trust that members of this Society will be alive to this danger which possibly exists in the future, and will strongly resist any attempt to reduce their status to that of an analytical machine.

In conclusion I would express the pleasure it has given me to preside, however imperfectly, over your deliberations and proceedings this year, and to acknowledge, as I heartily do, the help given me by the past Presidents, members of the Council, and, not least, by the very efficient Secretaries of the Society, Mr. Bevan and Mr. Chapman.

SOME NOTES ON THE ANALYSIS OF DRUGS.

By Sir Charles A. Cameron, C.B., M.D.

Determination of Alcohol in the Tincture and Solution of Iodine (B.P.).—In the *Pharmaceutical Journal*, 1898, ii., 333, it is recommended to fix the iodine with potash before distilling the tincture or solution. I find that the potash often causes "bumping," and there is some danger that an excess might be used which would act upon the alcohol. The addition of iron turnings to the tincture rapidly fixes the iodine, and is an improvement on the potash method.

Syrup of Ferrous Phosphate with Quinine and Strychnine.—The small quantity of strychnine and the large amount of quinine in this syrup render the estimation of the former a difficult operation, especially as only small quantities of the syrup are usually sent for analysis. I find the following method satisfactory: Both alkaloids are precipitated together and weighed together. The sulphuric acid is then determined and calculated as sulphate of quinine; the excess of alkaloid is strychnine. In 100c.c. of this syrup there are 14·8 grammes of quinine sulphate, and only 0·57 gramme of strychnine.

Tincture of Assafœtida.—In Gadd's useful little book, "A Synopsis of the Pharmacopœia," fifth edition, it is stated that tincture of assafœtida contains 10 per

cent. of solid matter in solution. In specially-prepared tincture I find that the solid matter is only 5 grammes per 100 c.c. The Pharmacopœia states that assafœtida should dissolve to at least 62 per cent. in spirit of 90 per cent. alcohol. It would have been better if its solubility in spirit of 70 per cent. alcohol had been given, as that spirit is the one employed in the preparation of the tincture. As the tincture is prepared by acting upon 200 grammes of the gum with 1,000 c.c. of spirit of 70 per cent. alcohol, one might naturally think that the spirit would hold about 10 per cent. of solid matter in solution; but this, as I have stated, is not the case. When the tincture containing 5 grammes of solids per 100 c.c. is mixed with 5 per cent. of water, it becomes clouded.

Tincture of Nux Vomica.—In Gadd's book it is stated that this tincture contains 45 per cent. of alcohol, but if it is prepared according to the Pharmacopœia it should contain 52·2 per cent. of absolute alcohol by volume.

Solution of Ferric Chloride.—The Pharmacopœia gives the specific gravity of liquor ferri perchloridi as 1·11. It is prepared by diluting the strong solution of ferric chloride, and as its strength is stated to be the same as that of the tincture of ferric chloride, it should contain 5·625 grammes of iron per 100 c.c. I find, however, that unless the specific gravity is greater than 1·11 it does not contain the supposed amount of iron.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Estimation of Fusel Oil in Brandy or other Alcoholic Liquors. E. Beckmann. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 1059-1064.)—The following method was found by the author to give satisfactory results: 50 c.c. of the brandy, together with 20 grammes of pure granular calcium chloride, are placed in a separating funnel, and so much water is added that the alcohol in the solution shall not exceed 50 per cent. When the calcium chloride has dissolved, the solution is thoroughly well extracted with four successive quantities of about 30 c.c. each of carbon tetrachloride. These extractions are collected in another separating funnel, and shaken with 25 c.c. of water to remove ethyl alcohol. The water may then be shaken with 80 grammes of carbon tetrachloride, after the addition of 10 grammes of calcium chloride. The total amount of carbon tetrachloride is dried by means of a little fused calcium chloride, and filtered through a plug of glass wool into a stoppered flask, the glass wool being washed with a little dry carbon tetrachloride. Three grammes of powdered acid sodium sulphate and 3 grammes of sodium nitrite are then added, and the flask is repeatedly shaken for thirty minutes. The solution is again filtered through glass wool, and the saline residue washed with a little

carbon tetrachloride. The filtrate is collected in a stoppered flask, and 3 grammes of powdered acid sodium carbonate are added. This removes the excess of nitrous acid. Water is added after the evolution of carbon dioxide has ceased, and the solution of the esters in the carbon tetrachloride is drawn off by means of a separating funnel. Ten c.c. of concentrated sulphuric acid are then added to decompose the esters and the mixture well shaken; 100 to 170 c.c. of ice-water containing pieces of ice are then added, and the solution is titrated with a solution of potassium permanganate (1 gramme per litre). Aldehydes, when present, interfere with the accuracy of the results, and may be removed by shaking the carbon tetrachloride solution with acid sodium sulphite and washing out the aldehyde compound formed with water. As this may introduce traces of sulphurous acid, the nitrite should be estimated in a nitrometer. W. P. S.

Detection of Heated Milk. M. Siegfeld. (*Milch Zeit.*, 1901, xxx., 723; through *Chem. Zeit. Rep.*, 1901, 366.)—The author has investigated Storch's *p*-phenylenediamine test for raw milk, and he finds that the temperature of heating which causes that reaction to fail is between 71° and 80° C., being chiefly dependent upon the acidity of the milk, and partly upon the time over which the heating extended. Therefore the test affords no method of discovering whether the milk has been raised to any temperature exceeding 80°. Still, it is so delicate that it will show the presence of 10 per cent. of raw milk in heated milk. The best method of carrying out the test is to shake about 10 c.c. of the sample with 1 or 2 drops of "medicinal" hydrogen peroxide, then adding 2 or 3 drops of a 2 per cent. alcoholic solution of *p*-phenylenediamine. A bluish colour will develop in the heated-milk tube on exposure to air and light, but the tint only becomes pronounced after several hours.

See Analyst 1903, 184— F. H. L.

Detection of Heated Milk. Du Roi and Koehler. (*Der Landbote*,* No. 103, 1901; through *Chem. Zeit. Rep.*, 1902, 13.)—Fifty c.c. of the milk are shaken with 1 c.c. of 1 per cent. hydrogen peroxide, and 3 c.c. of the mixture are poured into a tube containing an equal volume of a solution of starch and potassium iodide. If the milk is raw, thorough agitation causes the appearance of the blue iodine colour; but with heated milk the mixed liquids remain colourless. The test will show 2 per cent. of raw milk in heated milk. Potassium bichromate and formaldehyde, used in the ordinary proportions as preservatives, do not interfere. Raw sour milk yields the blue colour immediately. The critical temperature is about 80° C.

F. H. L.

Notes on the Estimation of Phosphoric Acid in Milk, etc. F. Rieger. (*Zeits. physiol. Chem.*, 1901, xxxiv., 109; through *Chem. Zeit. Rep.*, 1901, 366.)—In order to obtain an ash perfectly free from carbon, 50 c.c. of the milk should be evaporated to a syrup in a large platinum basin, stirring continuously, and finally adding some dry, powdered sodium carbonate. The residue is carbonized and ignited

* This is the organ of the Landwirthschaftskammer for the province of Brandenburg.

for fifteen minutes, and then heated once again, with agitation, under a layer of 1 part of sodium carbonate and 2 of potassium nitrate, until it becomes pasty. The product will be found to dissolve in cold nitric acid to an almost absolutely clear liquid.

For the estimation of total phosphorus in milk the sample may be precipitated with sodium hydroxide and copper sulphate solution, as in Ritthausen's process for the determination of albuminoids. The precipitate, which contains all the phosphorus as well as the albuminoids, is then incinerated with sodium carbonate and potassium nitrate as above described. F. H. L.

So-called Honey Dextrin. E. Beckmann. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 1065-1069.)—When 5 c.c. of a 20 per cent. solution of pure coniferous honey are mixed with 3 c.c. of a 2 per cent. solution of barium hydroxide and 17 c.c. of methyl alcohol, little, if any, precipitate is obtained. Should the honey, however, contain starch syrup, a precipitate is formed. This shows that the dextrin of coniferous honey is different to that present in starch syrup. The author has investigated the behaviour of the different dextrins of commerce, and finds that they give the following barium precipitates when treated as above :

A 20 per cent. Solution of :	Colour given with Iodine Solution.	Barium Precipitate.
Dextrin puriss. Merck	Cherry red	104·9 per cent.
,, technic. Merck	Dark blue	91·5 ,,
,, granular. Merck	Cherry red	86·45 ,,
,, Pharm. Germ. I. Merck ...	,, ,,	78·4 ,,
,, from starch syrup	No colour	41·3 ,,
,, from starch sugar	,,	29·3 ,,
,, from honey	,,	8·18 ,,

E. Prior found that achroodextrin III., giving 44 per cent. of barium precipitate, had a molecular weight of 642, and the formula $2(C_{12}H_{20}O_{10}) + H_2O$. Honey dextrin has apparently a still lower molecular weight.

Similar results to the above were obtained by using basic lead acetate in place of barium hydroxide, the yield of precipitate being somewhat larger with the former. The sugars in honey to some extent prevent the precipitation of dextrin, so that small quantities of added starch syrup may not be detected by this test.

J. Monheim ascertained that the benzoyl esters of honey dextrin correspond to a mixture of penta- and hexabenzoate of a disaccharide. Like the benzoates of saccharose and maltose, these compounds of honey dextrin are soluble in ether, whilst the benzoate of ordinary starch dextrin was almost insoluble. The benzol sulphonic esters obtained from honey dextrin corresponded with a disaccharide with 4 to 6 acid radicals, the *m*-nitro-benzoate to a saccharide with 5 to 6 acid radicals.

W. P. S.

The Detection of Artificial Colouring Matters in Sausages. E. Spaeth. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 1020-1023.)—The finely-divided sausage is heated for some hours at 100° C. and then extracted with ether. The fat-free substance is now warmed on the water-bath with a 5 per cent. solution of sodium salicylate for one hour. The coloured solution (if colouring matter be present) is filtered, acidified with sulphuric acid, and heated with some wool free from fat. It is not necessary to remove the precipitated salicylic acid by means of ether, etc., as it re-dissolves on warming, and in no way interferes with the fixing of the colour by the wool. The dyed wool may then be used for the identification of the colour. W. P. S.

The Adulteration of Pepper with the Fruit of Myrsine Africana and Embelia Ribes. A. Mennechet. (*Journ. Pharm. Chim.*, 1901, xiv., 557-561.)—The adulteration of pepper with these substances is still practised, though, according to the author, less frequently since 1889.

After giving a description of the structure of the two fruits, as seen under the microscope, the author points out that it would not be easy to identify a small addition of either of them to pepper by this means, and he therefore recommends the following chemical test: The powder is lixiviated with ether, and the yellow extract shaken with several times its volume of water rendered alkaline with ammonia. The lilac-red colouring matter (ammonium embelate) formed is insoluble in ether, but is dissolved by the water. On the addition of an acid the red colour disappears, and the embelic acid again dissolves in the ether.

Myrsine, which gives the same reactions, appears to contain a closely-related compound.

No coloration is given by pure pepper when thus treated. C. A. M.

The Official Test for Myrrh. H. G. Greenish. (*Pharm. Journ.*, 1901, 666.)—As an improvement on the official test, the following is recommended: Half a gramme of coarsely-powdered myrrh, occasionally shaken during ten minutes, with 10 c.c. of ether, should afford a filtrate, 2 c.c. of which should yield, when evaporated, a residue that is slowly coloured violet by contact with the vapour of nitric acid. Bissabol, bdellium, hotai, and other gums, do not give any violet reaction. W. P. S.

Notes on the Estimation of Berberine. H. M. Gordin. (*Amer. Journ. Pharm.*, 1902, lxxiv., 37-39.)—In the author's first method of determining berberine (*Arch. d. Pharm.*, 1901, 638; *cf.* ANALYST, xxiv., 238), the alkaloid is extracted from the drug by means of hot alcohol, the solution made up to a definite volume with cold alcohol, and the berberine precipitated as an acid sulphate which is subsequently converted into the mono-acid hydriodide.

Recent experiments have shown that although berberine in the form in which it occurs in *Hydrastis canadensis* and some other plants is readily soluble in alcohol,

yet in others, such as barberry bark, it is frequently present in a form which only dissolves with difficulty in cold alcohol; so that on adding cold alcohol to the hot alcoholic extract some of the berberine is precipitated.

To guard against this the sediment should be dissolved in water, filtered, and the clear filtrate tested for berberine. If present, it should be determined by the second method described in the former paper (*loc. cit.*).

As regards the precipitation of berberine by potassium iodide, the author states that in the presence of free acid Mayer's or Wagner's reagents effect more complete precipitation, and it is therefore advisable to use Mayer's reagent in the first method, in which acid is liberated in the reaction.

A further improvement is to collect the berberine acid sulphate upon a plug of cotton-wool in a filter, and to wash the beaker out twice with 5 c.c. of a mixture of alcohol and ether (1 : 1), and then to wash the precipitate with ether, in which it is completely insoluble. The amount of berberine in the 10 c.c. of alcohol-ether washing is less than 0·0006 gramme, and may be neglected. C. A. M.

TOXICOLOGICAL ANALYSIS.

The Detection of Free Phosphorus in the Presence of Alcohol. J. Habermann and A. Oesterreicher. (*Zeit. anal. Chem.*, 1901, xl., 761-766.)—Various substances, and in particular alcohol, interfere with the production of the phosphorescence in Mitscherlich's method of detecting phosphorus (*cf.* ANALYST, xxiii., 39). According to the results of experiments made by the authors, this difficulty is avoided by the dilution of the alcoholic vapour or distillate with a sufficient quantity of water; and on this fact are based the following modifications of Mitscherlich's process:

1. The liquid is distilled in a dark room, and as soon as the steam is felt by the hand to have reached the upper part of the condenser, water is introduced through a stopcock.

2. The liquid is fractionally distilled in daylight, and the several fractions diluted with water in the dark. C. A. M.

Detection of Phosphorus in Cases of Poisoning. C. Binda. (*Giorn. Farm. Trieste*, 1901, vi., 225; through *Chem. Zeit. Rep.*, 1901, 354.)—It is known that certain liquids, like ethyl alcohol and turpentine, prevent the vapours of phosphorus from being visible in the dark. In such cases the author finds luminosity to occur if the liquid is evaporated rapidly on a glass plate in the dark, the plate being rubbed with a glass rod meanwhile. F. H. L.

The Alleged Poisonous Properties of the Colours "Mandarin" and "Metanil Yellow." J. Frentzel. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 968-974.)—In this paper are given the results of a considerable number of

experiments, consisting in feeding rabbits, dogs, and human beings with food mixed with the colours "mandarin" (obtained by diazotising sulphanilic acid and β-naphthol) and "metanil yellow" (prepared by diazotising meta-benzene-sulphonic acid and diphenylamine). The conclusions arrived at are that the colours can scarcely be considered poisonous in the small quantities in which they are used in foods, etc. Long-continued, large doses, however, give rise to some injurious effects; but this quantity is never, in the natural course of things, even approximately reached. W. P. S.

ORGANIC ANALYSIS.

Gravimetric Determination of Formaldehyde. L. Vanino. (*Zeit. anal. Chem.*, 1901, xl., 720, 721.)—This method is based upon the facts that, on treating a solution of formaldehyde with silver nitrate and sodium hydroxide, a mixture of metallic silver and silver oxide separates, and that the silver oxide is dissolved on adding dilute acetic acid to the mixture. Thus:

$$4AgNO_3 + 4NaOH = 2Ag_2O + 4NaNO_3 + 2H_2O$$
$$2Ag_2O + 2NaOH + 2HCOH = 2HCOONa + 4Ag + 2H_2O.$$

In making a determination an aqueous solution of 2 grammes of silver nitrate is rendered strongly alkaline with sodium hydroxide, 5 c.c. of a 10 per cent. solution of commercial formalin introduced with continual stirring, and the whole put aside in a place protected from the light.

After fifteen minutes the clear supernatant liquid is poured off through a weighed filter and the precipitate digested three or four times with 5 per cent. acetic acid, transferred to the same filter, washed with water rendered slightly acid with acetic acid until free from chlorine, dried at 105° C. and weighed.

The results quoted are very concordant and in close agreement with those obtained by other methods. C. A. M.

On the Separation of Terpene Alcohols from their Esters by means of Sodium Salicylate. G. Darzens and P. Armingeat. (*Bull. Soc. Chim.*, 1901, xxv., 1053-1055.)—Charabot and Hébert have recently described a method of separating terpene alcohols from their esters by treating the mixture with a 50 per cent. solution of sodium salicylate, in which only the alcohols were said to be soluble. (*Bull. scient. et indust. de la Maison Roure-Bertrand*, October 5, 1901; *cf.* ANALYST, xxv., 72, 188.)

The authors have made experiments with this method, and have come to the conclusion that little reliance can be placed upon it as a quantitative method. Mixtures of linalol, geraniol, and rhodinol with their respective esters were prepared and the saponification values of the mixtures determined. They were then treated four times with an equal volume of 50 per cent. sodium salicylate solution, and after standing for twenty-four hours the insoluble portion was decanted and treated

with water in excess. Finally, the saponification value of each of these fractions was determined.

The following table of results thus obtained shows that in each case the dissolved portion contained considerable quantities of esters.

	Mixture.		Insoluble Portion.		Soluble Portion.	
	Saponification Value.	Esters per Cent.	Saponification Value.	Esters per Cent.	Saponification Value.	Esters per Cent.
Geraniol and geranyl acetate	116	40·6	110	38·5	120·8	42·3
Linalol and linalyl acetate	98·2	34·4	116·3	40·7	85	29
Rhodinol and rhodinyl acetate	126	45	Mixture completely soluble			

C. A. M.

A New Colour Reaction for Cholesterol. L. Tschugaew. (*Jour. russ. phys.-chem. Gesellsch.*, 1900, xxxii., 363; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 974, 975.)—The test consists in dissolving cholesterol in glacial acetic acid, and adding an excess of acetyl chloride together with a small piece of zinc chloride. On warming, a red or rose coloration is produced, which also shows a greenish-yellow fluorescence. The maximum depth of colour is reached after five minutes' boiling. The test will detect 1 : 80,000. W. P. S.

The Detection of Vegetable Oils in Animal Fats by Means of the Phytosterol Acetate Test. A. Bömer. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 1070-1095.)—The method of carrying out this test is as follows: From 100 grammes of fat the crude cholesterol and phytosterol are obtained in the usual manner by shaking out the saponified fat with ether. The ethereal solution is well washed with water to remove dissolved soap, and the greater part of the ether is distilled off. The remainder of the ether is then allowed to completely evaporate in a small basin; the residue is taken up in the least possible quantity of alcohol, and allowed to crystallize. These crystals may be examined microscopically to see whether cholesterol or phytosterol be present. The alcohol is then completely driven off on the water-bath, 2 to 3 c.c. of acetic anhydride are added, and the contents of the basin boiled for a quarter of a minute over a wire-gauze, the basin being covered with a clock-glass. The excess of acetic anhydride is evaporated on the water-bath. Sufficient absolute alcohol is now added to just dissolve the esters on heating. To prevent immediate crystallization, 1 c.c. more of absolute alcohol is added, and the esters allowed to crystallize at the ordinary temperature of the room. After one-half to one-third of the alcohol has evaporated and the greater part of the esters have crystallized out, the crystals are filtered through a small filter, again

dissolved in alcohol, and allowed to crystallize. This recrystallization is repeated as many times as the quantity of the crystals will allow. The melting-point of the crystals is taken after the third and subsequent crystallizations. Should the melting-point of the crystals be 116° C., the presence of vegetable oil may be suspected; if the melting-point be 117° C., or higher than this, vegetable oil is certainly present. The melting-point should be corrected by the formula $S = T + n(T - t) \times 0{\cdot}000154$, where S is the correct melting-point, T the observed melting-point, n the length of the column of mercury above the surface of the liquid, and t the average temperature of the column of mercury outside the liquid. This latter figure is obtained by means of a second thermometer placed at the middle of the column. W. P. S.

Note on Halphen's Test for Cotton-seed Oil. P. Soltsien. (*Zeit. öffentl. Chem.*, 1901, vii., 140-143; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 979.)—The author draws attention to the fact that American lards obtained from pigs fed on cotton-seed meal show strong reactions with Halphen's and Bechi's tests. The same lards, however, give no reaction with Welman's test, and phytosterin cannot be detected in them. The presence of added cotton-seed oil in lard is, therefore, only to be detected with certainty when confirmed by the two latter tests. W. P. S.

Formula for the Calculation of Diglycerides in Fat Mixtures which contain Oxy-fatty Acids. J. Freundlich. (*Chem. Zeit.*, 1901, xxv., 1129.)—The formula given by Benedikt for this purpose is inconvenient, as it requires two acetylations—that of the original fat and that of the fatty acids. Calling the ether value of the fat *d*, the yield of total fatty acids F, and the molecular weight of the diglycerides M, their percentage amount is given by the formula

$$D = M \times \frac{1683(100 - F) - 38d}{51612}.$$

If no soluble fatty acids are present, F may be replaced by the Hehner number; otherwise that figure must be ascertained analytically. Presence or absence of oxy-fatty acids is immaterial, and they may exist either as neutral fats or in the free state. The steps involved in arriving at the above formula are explained in the original article. Freundlich remarks that both in the *Zeits. angew. Chem.*, 1888, 461, and in Benedikt's book (3rd edition, p. 165), there is an error in the corresponding formula quoted, the first figure in the denominator being given as "5600," instead of "56100." F. H. L.

Detection of Gallic Acid in Tanning Materials. M. Spica. (*Gazz. Chim. Ital.*, 1901, xxxi. [2], 201; through *Chem. Zeit. Rep.*, 1902, 4.)—The reagent is a solution of potassium plumbite prepared by dissolving in the necessary quantity (not an excess) of potassium hydroxide the precipitate obtained therewith from a solution of lead acetate. On adding some of this liquid to a dilute solution of gallic acid, shaking repeatedly so as to encourage oxidation, and finally diluting with water, a

scarlet-red colour will be produced—a reaction which is neither given nor hindered by the presence of tannin. The method is capable of being made quantitative by colorimetry. F. H. L.

The Adulteration of Shell-lac. K. Dieterich. (*Chem. Revue ii. Fett und Harz-Ind.*, 1901, viii., 222-226 and 244-246.)—The results of a number of experiments with pure shell-lacs and mixtures of shell-lac and colophony lead to the conclusion that the determinations of the acid, ester, and saponification values are of little use for the detection of adulteration. The values obtained with mixtures still lay within the wide limits yielded by pure shell-lacs. Better results were obtained by determining the solubility of the samples in ordinary solvents, such as ether, petroleum spirit, benzol, etc. In ether colophony is insoluble, whilst shell-lac dissolves to the extent of 14 per cent. W. P. S.

Colour Reactions of Certain Coal-Tar Derivatives and Morphine with Formaldehyde and Sulphuric Acid. R. A. Hatcher. (*Amer. Journ. Pharm.*, 1902, lxxiv., 35, 36.)—The author's experiments have led him to the conclusion that the formaldehyde sulphuric acid test for morphine is a reliable one; but, at the same time, care should be taken to distinguish the colour from the colorations given by the other compounds mentioned.

It was found that, in the case of mixtures of phenol and morphine, a large proportion of the former could be expelled, without decomposing the alkaloid, by evaporating an alkaline solution with strong sulphuric acid.

In the tests tabulated below 1 drop of the respective solutions was added to 5 drops of the reagent prepared by mixing 5 c.c. of formaldehyde (40 per cent.) with 100 c.c. of sulphuric acid, the following results being obtained:

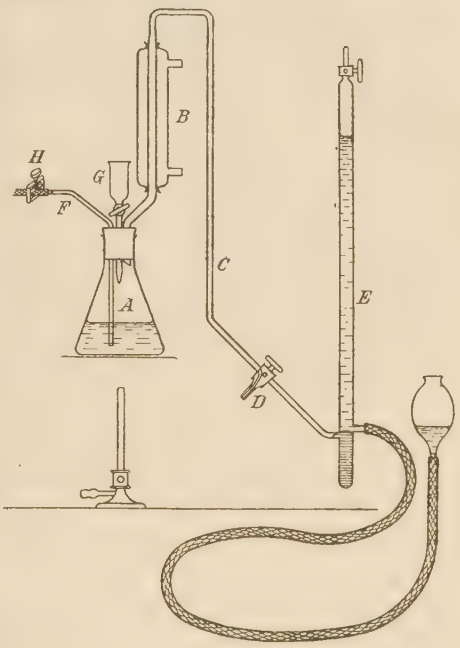
Phenol, 0·1 gramme + 5 c.c. $\frac{N}{10}$ NaOH, warmed and mixed with 10 c.c. $\frac{N}{10}$ H_2SO_4.	Morphine Sulphate, 0·01 gramme +5 c.c. $\frac{N}{10}$ NaOH +10 c.c. $\frac{N}{10}$ H_2SO_4.	Phenol, 0·1 gramme; Morphine Sulphate, 0·01 gramme + 5 c.c. $\frac{N}{10}$ NaOH + 10 c.c. $\frac{N}{10}$ H_2SO_4.	Sodium Salicylate (commercial), 0·1 gramme + 50 c.c. $\frac{N}{10}$ H_2SO_4.	Sodium Salicylate (Chem. Pure).	Salicylic Acid (Chem. Pure) dissolved in Alcohol.
Cold. Crimson to cherry, deepening to garnet on standing.	*Cold.* Maroon, deepening on standing.	*Cold.* Deep crimson, inclining to garnet.	*Cold.* *Faint* pinkish salmon.	*Cold.* None.	*Cold.* None.
On Heating. Maroon precipitate.	*On Heating.* Fades to dirty brown.	*On Heating.* Brown, maroon tint.	*On Heating.* Rose-pink.	*On Heating.* None, except fleeting rose-pink on superheated edges of vessel.	*On Heating.* Same as sodium salicylate. Odour of oil of wintergreen.

It is advisable, in the presence of these substances, to remove them from the solution by extraction with ether after acidification, and to apply the test for morphine to the residual solution. C. A. M.

Gasometric Estimation of Nitrites in Urine. P. Gerlinger. (*Zeits. f. angew. Chem.*, xiv., 1250.)—The nitrite by double decomposition with ammonium chloride is converted into ammonium nitrite, which is decomposed by boiling according to the equation :

$$NH_4NO_2 = 2H_2O + N_2.$$

In the flask *A* is placed a saturated neutral solution of ammonium chloride which has previousiy been boiled. Carbon dioxide is led into the apparatus through the tube *F* to entirely displace air, the liquid being meanwhile heated. The flame is now removed. The cocks *D* and *H* are closed, and the urine or other substance to be examined is introduced carefully through the funnel *G*. The liquid is now gently boiled, and all the nitrogen is driven by means of a current of carbon dioxide into the nitrometer *E*, and measured in the usual way. The author finds that the results are accurate. A. M.



Jolles' Process for the Estimation of Uric Acid in Urine. O. Makowka. (*Chem. Zeit.*, 1901, xxv., 1159.)—The present author has investigated this process, and finds it to be convenient and exact, while its simplicity, in comparison with the gravimetric method or the volumetric process of Hopkins and Folin, renders it preferable for practical purposes. Jolles' process is best carried out as follows: 100 c.c. of urine, previously warmed to 60° C. and filtered if turbid, are mixed with 10 grammes of solid ammonium acetate (or sulphate) in a 600 c.c. flask, and from 3 to 5 c.c. of strong ammonia are added with constant stirring till the odour is persistent. The whole is allowed to rest for four hours, the clear liquid run off through a filter, and the deposit of phosphates and uric acid washed by decantation some nine times with 10 c.c. of 10 per cent. ammonium carbonate solution till the filtrate no longer gives a chlorine reaction. The precipitate is next rinsed into a beaker with water at 80°, the liquid adjusted to 50 or 70 c.c., and the mixture boiled for about thirty or forty-five minutes with 0·1 to 0·2 gramme of pure magnesia until litmus-paper shows that all ammonium salts have been decomposed. It is then acidulated with weak sulphuric acid, diluted to 200 or 300 c.c., and treated with 10 c.c. of 50 per cent. sulphuric acid (specific gravity 1·4). The whole is gently boiled, and a solution of potassium permanganate (8 grammes per litre) is run in, 1 c.c. at a time at first, afterwards in quantities of 6 drops, till the pink colour withstands fifteen or twenty minutes' boiling, the volume of the liquid being not less than 100 c.c. Finally it is concentrated to 60 c.c., when decolorization usually occurs, and

a part of the urea is converted into ammonia—but this is of no consequence; if the liquid is not quite clear and colourless, a minute amount of oxalic acid is added. The product is transferred to a vessel which can be cooled, where it is treated with 33 per cent. sodium hydroxide solution, 1 c.c. at a time, until it is alkaline to litmus, and a flocculent precipitate begins to appear, taking care that the temperature does not exceed 15° or 20° C. In a suitable nitrometer, such as that which Jolles has described, this liquid is decomposed with 50 c.c. of a solution containing 80 grammes of sodium hydroxide and 25 grammes of bromine per litre, the evolved nitrogen being corrected and converted into weight by means of the usual tables.

Makowka quotes a series of six normal and pathological urines analysed by the Ludwig-Salkowski and by Jolles' method. The figures show that the latter gives results from 0·8 to 3·1 (mean 2·1) per cent. higher than the former. Inasmuch as the Ludwig-Salkowski process when tested on pure uric acid yields 2 per cent. too little, it follows that Jolles' method is accurate. F. H. L.

A Gold Reagent for Colloidal Substances. R. Zsigmondy. (*Zeit. anal. Chem.*, 1901, xl., 697-719.)—The author's reagent is prepared by heating 120 c.c. of water (distilled through a silver condenser) to the boiling-point, and adding 2·5 c.c. of a solution of gold hydrochloride (6 grammes $AuCl_3.HCl$, $3H_2O$ per litre) and 3 to 3·5 c.c. of a solution of pure potassium carbonate (0·18 N.). Immediately after the liquid boils 3 to 5 c.c. of a dilute solution of formaldehyde (0·3 c.c. of formalin in 100 c.c.) are added with vigorous stirring, with the result that a deep red permanent colour is produced.

On mixing 5 to 10 c.c. of this solution with a few drops of a solution of a colloid and adding 0·5 to 1 c.c. of a 10 per cent. solution of sodium chloride, a difference is observed with different classes of colloidal substances.

The author classifies them in the following groups according to their behaviour with the reagent:

1. *Active Colloids.*—Glue, gelatin, casein, isinglass, etc. From 0·005 to 0·1 milligramme of these is sufficient to prevent the change of colour of 10 c.c. of a 0·0056 per cent. gold solution to violet or blue after the addition of 1 c.c. of sodium chloride solution.

2. Gum-arabic, egg albumin, gum tragacanth, carrageen, etc. These are less active, from 0·1 to 10 milligrammes being required to prevent the change of colour.

3. Dextrin, potato starch, etc., of which from 10 to 500 milligrammes are required.

4. *Inactive Colloids.*—To this group belong silicic acid, soluble glass, and other substances, the addition of which does not interfere with the change of colour. On long keeping, however, the restrictive power of most colloidal solutions shows a marked decrease.

The author applies the term *gold value* to the number of milligrammes of a colloid necessary to prevent the change of colour of 10 c.c. of the gold reagent immediately or soon after the addition of 1 c.c. of sodium chloride solution.

He has obtained the following gold values for different colloids: Freshly prepared

glue and gelatin solutions, 0·005 to 0·01 ; gum arabic, 0·1 to 2 or 4 ; dextrin, about 20 ; and colloids of the fourth class, nil.

It is pointed out that well-prepared gold solutions undergo changes when kept for a long time, and that under such circumstances they should be standardized on gum arabic or dextrin, the gold values of which are known.

The effect of various factors on the gold value is also discussed. C. A. M.

INORGANIC ANALYSIS.

Determination of Silver in the Residues from the Distillation of Zinc. K. Sander. (*Zeits. f. angew. Chem.*, xv., 32.)—The residues contain a considerable quantity of lead, 20 or 30 per cent. of carbon, and all the silver that was in the ore. In order to remove the carbon rapidly the author oxidizes with a mixture of 80 parts potassium nitrate and 20 parts sodium peroxide. He mixes 20 grammes of the coarsely-powdered residue with 50 grammes of the above mixture, and introduces it in small quantities of 3 or 4 grammes at a time into a red-hot iron crucible. As soon as the reaction is not very violent the crucible is placed in the fire, the necessary quantity of flux is added, and 10 grammes of litharge. The button of lead obtained is cupelled in the usual way. A. M.

The Separation of Copper, Lead, Antimony, and Tin in Alloys. A. Rössing. (*Zeit. anal. Chem.*, 1902, xli., 1-11.)—*Solubility of Copper Sulphide in Alkali Sulphides.*—The author shows in a series of experiments that copper sulphide is soluble in yellow ammonium sulphide and in sodium polysulphide, the respective compounds $(NH_4)_2Cu_2S_7$ and $Na_2Cu_2S_7$ being formed. The loss in the analysis of alloys is greater when the influence of the air is excluded, and in some of the experiments amounted to as much as 5·71 per cent. of the total amount of copper.

On the other hand, accurate results were obtained by using *colourless* sodium sulphide, and the author therefore considers it essential to use that compound in the separation of metals of the hydrogen sulphide group when copper is present.

Thus, since only the alkali monosulphide is applicable for the separation of copper from tin, antimony, and arsenic, care must be taken that the tin has been sufficiently oxidized or chlorinated.

Separation of Lead and Copper from Tin and Antimony.—The method of converting the tin and antimony into insoluble oxides by treatment with nitric acid, whilst converting the lead and copper into soluble nitrates, has not yielded good results in the author's hands. In every instance he found more or less copper and lead in the insoluble residue.

The following method is recommended : Two grammes of the coarsely-divided alloy are dissolved in the smallest possible amount of *aqua regia*, and the solution heated with a little potassium chlorate as a precaution. It is then diluted with water, a little tartaric acid being added, rendered slightly alkaline with sodium hydroxide, and treated with the smallest possible excess of colourless sodium

sulphide. The liquid is heated for a short time, allowed to stand, and filtered. The residue is washed with water containing a little sodium sulphide, and dissolved in hot dilute nitric acid, and the lead and copper in the solution separated in the usual way.

Separation of Tin from Antimony.—Von Carnot's method, which is based upon the precipitation of the antimony as oxysulphide by sodium thiosulphate,

$$2SbCl_3 + 3Na_2S_2O_3 = Sb_2OS_2 + 6NaCl + 4SO_2,$$

has given inaccurate results in the author's experiments, as was also the case with the modification proposed by Andrews (*Zeit. anal. Chem.*, xxxviii., 796).

Clarke's method, however (*Zeit. anal. Chem.*, ix., 487), which consists in precipitating the antimony from the boiling solution by means of hydrogen sulphide in the presence of much oxalic acid and absence of much free mineral acid, yielded accurate results.

In determining the antimony thus separated the author proceeds as follows: The precipitate is washed with hot water, and then successively with alcohol, a mixture of alcohol and carbon bisulphide (to remove free sulphur), alcohol, and finally ether, and is dried at a low temperature.

It is next detached from the paper, the small residual portion being dissolved off with hot ammonium sulphide, and ignited in a weighed porcelain crucible. The main portion is treated with concentrated nitric acid, which is evaporated on the water-bath, the oxidation being repeated until unoxidized sulphur can no longer be observed. The temperature is then raised, with the precautions noted by Brunck (ANALYST, xx., 190), and the crucible finally heated over the blowpipe until constant in weight. C. A. M.

Valuation of Commercial Aluminium Sulphate. C. R. Gyzander. (*Chem. News*, 1901, lxxxiv., 296 and 306.)—The sample is brought into solution, mixed with a small known quantity of standard sulphuric acid equivalent in strength to the alkali used, and titrated with sodium hydroxide (say of 11·65 grammes of NaOH per litre, which gives direct readings in percentages of Al_2O_3 when 0·5 gramme of sulphate is employed) in presence of methyl orange and phenolphthalein, till the colour changes from pink to orange, not yellow. According to the amount of alkali used, allowing for the acid purposely added, the sample is either basic, normal, or acid. Titration is then continued at 30° C. till a permanent pink is produced, and this gives the total alumina. The object of the methyl orange is to mask the pink tint of the phenolphthalein-alumina lake, which is otherwise troublesome.

Minute amounts of free acid, which cannot be determined by titration, can be estimated by observing the time taken for a solution of the aluminium sulphate to decolorize a "solution" (1 gramme per litre) of a standard sample of artificial ultramarine in comparison with that required by a pure sulphate, or by a specimen thereof to which known quantities of acid have been introduced. F. H. L.

Presence of Arsenic in Aqueous Ammonia. O. Gottheil. (*Pharm. Zeit.*, 1901, xlvi., 992; through *Chem. Zeit. Rep.*, 1901, 365.)—The author has found 0·057 per cent. of As_4O_6 in an ordinary commercial specimen of 25 per cent. ammonia.

F. H. L.

Determinations of the Strength of Aqueous Solutions of Hydrofluoric Acid. F. Winteler. (*Zeits. f. angew. Chem.*, xv., 33.)—All glass beakers, pipettes, rods, or other glass articles which may come in contact with the hydrofluoric acid must be well covered with paraffin wax. The acid should be titrated with standard caustic alkali, using phenolphthalein as indicator (see ANALYST, 1898, 165). The caustic alkali must, of course, be free from carbonate. Using these precautions the author has drawn up the following table of specific gravities of chemically-pure hydrofluoric acid. The figures differ considerably from those of Eckelt (ANALYST, 1898, 193).

SPECIFIC GRAVITIES AT 20° C.

Per cent. HF.	Specific Gravity.	Degrees Baumé.	Per cent. HF.	Specific Gravity.	Degrees Baumé.
1	1·003	0·5	26	1·092	12·0
2	1·007	1·0	27	1·095	12·4
3	1·011	1·5	28	1·098	12·7
4	1·014	2·0	29	1·101	13·1
5	1·018	2·5	30	1·104	13·4
6	1·023	3·0	31	1·106	13·7
7	1·027	3·5	32	1·109	14·1
8	1·030	4·0	33	1·112	14·4
9	1·035	4·5	34	1·114	14·7
10	1·038	5·0	35	1·117	15·0
11	1·041	5·5	36	1·120	15·4
12	1·045	6·0	37	1·122	15·7
13	1·049	6·5	38	1·125	16·0
14	1·052	7·0	39	1·127	16·3
15	1·055	7·5	40	1·130	16·5
16	1·059	8·0	41	1·133	16·8
17	1·062	8·4	42	1·136	17·2
18	1·066	8·8	43	1·138	17·5
19	1·069	9·3	44	1·141	17·8
20	1·072	9·7	45	1·143	18·1
21	1·076	10·1	46	1·146	18·4
22	1·079	10·5	47	1·149	18·7
23	1·082	10·9	48	1·152	19·0
24	1·086	11·3	49	1·154	19·3
25	1·089	11·7	50	1·157	19·5

A. M.

Colorimetric Estimation of Nitric Acid. H. Noll. (*Zeits. f. angew. Chem.*, xiv., 1317.)—After reviewing various methods which have been proposed for estimating nitric acid in water by means of brucine (*cf.* ANALYST, 1881, 56; 1901, 306) the author gives his own: To 10 c.c. of the water add 0·05 gramme of brucine

dissolved in 20 c.c. of sulphuric acid (specific gravity 1·840), stir for a quarter of a minute, then pour into a Hehner-Nessler tube which already contains 70 c.c. of water. For comparison a solution is made containing 0·1871 gramme of potassium nitrate per litre. Of this, 10 c.c. = 1 milligramme of nitric acid (N_2O_5). For the determination, 5 c.c. or less are taken, diluted to 10 c.c., and treated exactly as the water under examination, the colours being finally adjusted by running out some of the liquid which is the more strongly coloured. If the water under examination contain more than 50 milligrammes of nitric acid per litre it must be diluted. The results agree fairly well with those obtained by the methods of Marx, Trommsdorf, Schulze-Tiemann, and Henriet. A. M.

A Gas-Volumetric Method of Determining Free and Combined Sulphuric Acid, and Free and Combined Carbonic Acid. **E. Riegler.** (*Zeit. anal. Chem.*, 1902, xli., 17-25.)

Sulphates and Sulphuric Acid.—A hydrochloric acid solution of the sulphate is precipitated with a known excess of barium chloride solution (30·5 grammes per litre; 1 c.c. = 0·010 gramme SO_3), and the excess of barium chloride precipitated as iodate, which on treatment with hydrazine sulphate yields elementary nitrogen as a product of the reaction. Thus:

1. $BaCl_2 + 2HIO_3 = Ba(IO_3)_2 + 2HCl$.
2. $Ba(IO_3)_2 + 3N_2H_4H_2SO_4 = BaSO_4 + 2H_2SO_4 + 2HI + 6H_2O + 6N$.

The details of the method are as follows: From 30 to 40 c.c. of the solution (which should only contain a very small amount of sulphate) are acidified with 5 drops of concentrated hydrochloric acid, and the sulphate precipitated with 5 c.c. of the standard barium chloride solution, further additions of 5 c.c. each being made until the precipitation is complete. This is shown by testing a drop of the liquid with potassium chromate solution; and it is essential that the excess of chloride shall not exceed 5 c.c., in order to avoid the evolution of too large a volume of nitrogen.

The liquid is boiled, shaken, allowed to cool, shaken with 5 c.c. of a 6 per cent. solution of iodic acid, and left for fifteen minutes.

The precipitate of barium iodate and sulphate is now collected on a filter, and washed with water until the filtrate and washings amount to 100 c.c., after which the filter-paper is rolled up and placed in the inner vessel of a Knop-Wagner nitrometer. The outer vessel receives 40 c.c. of a 2 per cent. solution of hydrazine sulphate, and the nitrogen evolved is collected and reduced to standard pressure and temperature in the usual manner (*cf.* ANALYST, xxvi., 215, 239, and xxvii., 72).

One milligramme of nitrogen corresponds to 0·9504 milligramme of SO_3, or 1 c.c. of nitrogen at 0° C., and 760 millimetres pressure to 1·192 milligrammes of SO_3. A correction, experimentally found to be 0·0035 gramme, is made for the solubility of the barium iodate in the 100 c.c. of washings.

Free and Combined Carbonic Acid.—The method is based upon the conversion of barium carbonate into chloride, the formation of iodate from the chloride, and the liberation of nitrogen by the action of hydrazine sulphate on the iodate.

The solution of the carbonate, which must not contain more than 0·025 gramme of carbonic acid, is shaken with a few c.c. of a solution of barium hydroxide, and the resulting precipitate of barium carbonate collected, washed, and treated with about 30 c.c. of water and 15 drops of concentrated hydrochloric acid. After the flask has been well shaken, 5 c.c. of a 6 per cent. solution of iodic acid are introduced, the flask again shaken for fifteen seconds, and then allowed to stand for fifteen minutes.

The precipitated barium iodate is collected, washed, and treated with hydrazine sulphate as described above.

Each milligramme of nitrogen corresponds to 0·5223 milligramme of carbon dioxide, or 1 c.c. of nitrogen at 0° C., and 760 millimetres' pressure is equivalent to 0·6552 milligramme CO_2. The correction for the solubility of the barium iodate in the 100 c.c. of filtrate and washings is 2·1 milligrammes of carbon dioxide.

The experimental results quoted are in close agreement with the theoretical amounts. C. A. M.

The Direct Gravimetric Determination of Boric Acid. A. Partheil and J. A. Rose. (*Berichte*, 1901, xxxiv., 3611, 3612.) This method is based upon the solubility of boric acid in ether saturated with water, and on its non volatility when the ethereal solution is evaporated *in vacuo* over sulphuric acid.

The authors recommend the apparatus shown in the figure for the determination. The boric acid solution, which has been acidified with hydrochloric acid, is introduced into the spiral tube, but should not reach as far as the bulb. Freshly-rectified ether is next added in sufficient quantity to nearly fill the bulb, whilst 20 c.c. of ether are placed in the weighed flask below the extractor. The extraction is continued on a water-bath for about eighteen hours, and then for a further two hours with a second weighed flask, so as to insure that all boric acid has been removed.

The flask is placed in a vacuum desiccator over sulphuric acid, and the residue dried until constant in weight, and weighed as H_3BO_3. In this way a solution of boric acid containing 0·31015 gramme yielded 0·3105 and 0·31025 gramme, whilst a solution of borax gave 0·12425 gramme as against the theoretical amount 0·12408 gramme.

The authors state that this method is also applicable to the determination of boric acid in minerals and in food-products.

The absence of sulphuric acid, nitric acid, phosphoric acid, iron in large quantity, zinc chloride, or arsenious acid, must be insured, since these substances also dissolve in the ether.

The purity of the residue obtained can be tested by repeatedly evaporating it with pure methyl alcohol, and deducting the weight of any non-volatile substance. C. A. M.

The Determination of Small Quantities of Hydrogen Sulphide in Natural Waters. L. W. Winkler. (*Zeit. anal. Chem.*, 1901, xl., 772-774.)—If the water is first treated with a solution of sodium potassium tartrate to prevent the separation of magnesium or calcium carbonate, and then mixed with an alkaline solution of lead acetate, a brownish tint is produced, which can be compared with that given by a standard solution of sulphide under the same conditions.

In making a determination, 100 c.c. of the water are shaken with 5 c.c. of a reagent consisting of 25 grammes of sodium potassium tartrate, 5 grammes of sodium hydroxide, and 1 gramme of lead acetate in 100 c.c. of water.

One hundred c.c. of distilled water and 5 c.c. of the reagent are placed in a similar flask, and a dilute standard solution of ammonium thioarsenite run in from a burette until the colour of the liquid in the first flask is matched.

The standard solution is prepared by dissolving 0·0367 gramme of pure arsenic trisulphide in a few drops of ammonium hydroxide, and diluting the solution to 100 c.c. One c.c. of this solution corresponds to 0·1 c.c. of hydrogen sulphide at 0° C. and 760 millimetres' pressure.

If the amount of hydrogen sulphide in the water is less than 0·2 c.c. per litre, the coloration is too faint, and a larger quantity (500 to 1,000 c.c.) of the sample must be taken.

This method has the advantage that the results are not influenced by the presence of thiosulphates, which are usually present in sulphuretted mineral waters. Its accuracy is shown by a series of test experiments. C. A. M.

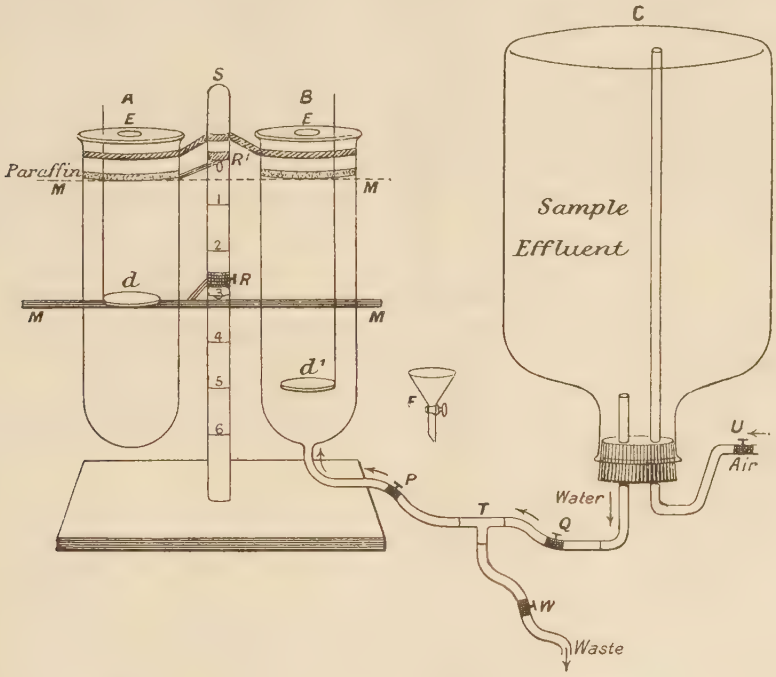
Colorimetric Method for determining Oxygen dissolved in Water. W. Ramsay and I. Homfray. (*Journ. Soc. Chem. Ind.*, xx., 1071.)—Use is made of the fact that dissolved oxygen oxidizes ammoniacal cuprous chloride solution to cupric chloride, the resulting blue colour being a measure of the oxygen present. The apparatus consists of two glass comparison tubes (*A*, *B*), 12 inches long and 2 inches in diameter; they are closed with movable caps (*E*), through which pass brass wires supporting the opal glass discs (*d*, *d*^{1}). The tubes are supported on a stand with a vertical brass stem (*S*), which is graduated in inches and tenths. The levels of the discs and of the liquids in the tubes are read off against the scale with the help of the horizontal levelling-rod (*M*, *M*), which slides along *S*.

Just before the experiment the bottle of effluent (*C*) is unstoppered and fitted with a rubber cork and tubes as shown. Some paraffin oil is poured into *B*, and the air is entirely displaced from the tubes by manipulating the screw-clips (*U*, *Q*, *W*, *P*). The effluent is allowed to run into *B*. Into *A* is poured distilled water of known temperature saturated with air. This also is covered with a layer of paraffin. Some powdered cuprous chloride is placed in the small tap-funnel (*F*), and covered with hydrochloric acid; a dark-brown solution results, some of which is run into *A* and *B*, avoiding air bubbles. Equal quantities of aqueous ammonia are then added, and the water is stirred gently by means of the discs. The discs are adjusted until, on looking at them through holes in the caps, they appear equally tinted. From

their position a simple calculation gives the percentage of saturation, and hence the number of c.c. of oxygen per litre in the effluent.

The cuprous chloride is made by warming a solution of cupric chloride with scraps of copper, pouring into water, filtering off, and washing with boiling water, alcohol, and ether. It should be quite white, and should be protected from light and damp in a stoppered black test-tube.

If much lime be present in the effluent, to prevent turbidity 2 or 3 c.c. of a hot saturated solution of ammonium chloride should be added to both tubes before the other reagents. If the effluent be coloured yellow a trace of alkaline paranitrophenol may be added to the standard water after the other reagents.



Portable Modification.—The test has been arranged in a portable form, all necessary sample-collecting apparatus, reagents and colour standards, being contained in a small wooden box. With this experiments can be carried out at the river-side in a few minutes. The standards of colour consist of six sealed tubes 6 inches long, containing various strengths of strongly ammoniacal cupric chloride. The experiment is carried out, under paraffin oil, in a similarly shaped tube with ground-glass stopper. Light is reflected up through the tubes by an opal glass plate set at 45°. The standards correspond with 1, 2, 3, 4, 5, 6 c.c. of oxygen per litre. It is easy to estimate within 0·5 c.c.

The results obtained by both modifications agree satisfactorily with those obtained by Thresh's method.

To avoid aeration in collecting samples, the can fitted to the testing-box is open

at one end, and has a valve opening inwards at the other. The stream can thus be allowed to flow right through, displacing all air. The presence of nitrites makes the results slightly high (see Rideal and Stewart, ANALYST, 1901, 143). A. M.

The Preparation of Sodium Arsenite Solution for Volumetric Analyses. G. Lunge. (*Zeit. f. angew. Chem.*, xiv., 1293.)—Petriciolli and Reuter in a footnote to a recent paper (*Ibid.*, xiv., 1181) stated that, if sodium arsenite be prepared by boiling arsenious anhydride with sodium bicarbonate, some sodium carbonate is formed which will itself absorb iodine. The author finds, however, that the quantity of iodine absorbed by carbonate under the conditions of an iodometric titration is inappreciable. A. M.

Preservation of Volumetric Solutions of Sodium Thiosulphate. F. H. Alcock. (*Pharm. Journ.*, 1901, 491).—Carbon bisulphide was found to act well as a preservative for standard solutions of sodium thiosulphate. Trials with a solution which was in use for a period of twelve months gave results justifying the recommendation. A. G. L.

The Use of Iron for Standardizing Permanganate Solution. H. Ort and W. Klaproth. (*Zeits. f. angew. Chem.*, xiv., 1233.)—Many samples of iron not very impure reduce considerably more permanganate than a chemically pure iron should. The following table shows the apparent percentage of iron calculated from the amount of permanganate solution reduced, the latter being standardized against pure oxalic acid :

Piano wire	99·41
Ferrum metallic. foliat.	100·31
Ferrum metallic. in lamin. pro analysi	100·55
Flower wire	100·07
Reduced iron	99·98

If 0·4 per cent. be added for the impurities in the iron, the results for most of the irons are still worse ; the error may then amount to 1 per cent. It is better to standardize with oxalic acid. A. M.

The Standardization of Uranium Solutions for Phosphate Analysis. J. A. Muller. (*Bull. Soc. Chim.*, 1901, xxv., 1000-1002.)—The uranium solution for the volumetric determination of phosphoric acid is frequently standardized on crystalline sodium phosphate, though the readiness with which this salt loses water is a source of error.

The crystalline salt (PO_4HNaNH_4, $4H_2O$) is much more stable ; but even that is less stable than the crystalline dicalcium phosphate—$(PO_4)_2Ca_2H_2$, $4H_2O$—which does not vary in weight when exposed to the air or when kept over phosphoric anhydride.

The author therefore recommends a solution of this salt in nitric acid as a suitable standard.

It is prepared by adding di-sodium phosphate little by little to a cold dilute solution of calcium chloride. The gelatinous precipitate soon becomes crystalline, and is then washed and dried on tiles at about 70° C. Theoretically it contains 41·27 per cent. of P_2O_5, whilst the author's preparations, analysed by different methods, were found to contain from 41·29 to 41·55 per cent. C. A. M.

The Solubility of Phosphatic Manures in some Organic Acids. Walter F. Sutherst. (*Chem. News*, 84, 199.)—In comparing the solvent action of organic acids on manures, the sample was extracted for twenty-four hours with solutions of the acids whose total acidity was the same in each case, and the dissolved phosphoric acid determined in a portion of the filtrates. The following table shows the percentages of the total tricalcic phosphate present which were found in solution:

Manure.	Acetic Acid.	Tartaric Acid.	Citric Acid.
Coprolite	... 12·01	43·41	20·36
Basic slag	... 42·22	54·41	67·50
Basic superphosphate ...	... 65·29	87·38	99·26
Precipitated phosphate ...	... 54·15	96·76	88·28

From his results the author concludes that the most satisfactory results on the whole are given by tartaric acid, and not by citric acid, the reagent generally employed. A. G. L.

Notes on some Blowpipe Tests. Joseph W. Richards. (*Journ. Am. Chem. Soc.*, xxiii., 213.)—Approximately quantitative determinations of water, volatile sulphur, etc., may be made by heating a weighed quantity of the material in a closed tube. The upper end of the tube containing the sublimate is broken off and weighed; in the case of water its ends are closed by small corks. The tube is then heated until the sublimate has been volatilized, and again weighed. A sample of göthite containing 10·11 per cent. of water gave 10·28 per cent. in this way.

When applying the flame test for phosphoric acid, the sample should be touched whilst hot with sulphuric acid, and then brought as slowly as possible to the outside edge of the Bunsen flame as low down as possible. For the detection of boron the sample should be held about an inch higher.

For the reduction of refractory minerals on charcoal with soda, it is always advantageous to add borax, especially in the case of tin dioxide.

In testing for fluorine by fusion with potassium bisulphate, Brazil wood paper is not reliable. The fusion is best made in a tube of 5 to 8 millimetres diameter, held nearly horizontal, when a silica ring deposits just above the assay. If the tube is broken off just under the ring, and the upper end held vertically under the nose, the odour of hydrofluoric acid can always be noticed.

To obtain sublimates from some arsenates, it is necessary to mix them with charcoal and soda, place this mixture in an open tube, and play on it with the reducing flame. All mercury compounds are also reduced in this way.

In testing for silica by means of the microcosmic bead test, it should be remembered that in the presence of certain oxides the bead is able to dissolve 30 per cent. of its weight of silica, and fairly large quantities of the sample may have to be taken. Some silicates dissolve as such in the bead, and in this case long blowing (five minutes) is necessary to decompose them and produce a skeleton of silica.

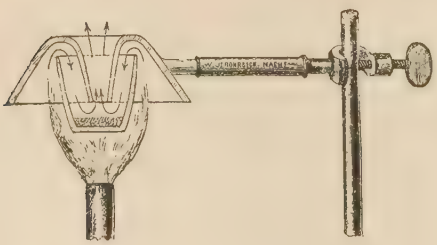
A. G. L.

Potassium Percarbonate as a Reagent. **F. P. Treadwell.** (*Chem. Zeit.*, 1901, xxv., 1008.)—Potassium percarbonate can now be procured commercially about 80 per cent. pure, and containing only small quantities of chloride and sulphate. As it keeps indefinitely in the dry state, it is a most useful substance from which to prepare hydrogen peroxide—by dissolving it in cold dilute acid—when wanted. The solution obtained by dissolving it in weak sulphuric acid is specially suitable in testing for titanium, vanadium, chromium, cerium, and for the oxidation of ferrous salts. Potassium percarbonate is also applicable in many cases where alkaline hydrogen peroxide is usually employed; it oxidizes sulphuretted hydrogen, soluble sulphides, and the lower sulphur acids to sulphuric acid; it converts manganese, nickel, and cobalt salts to higher oxides. It converts chromium salts into chromic acid, but its action here is not complete, like that of hydrogen peroxide. The percarbonate quickly reduces hypochlorites and hypoiodites to chlorides and iodides. The makers are the Neuhausen Aluminium Co. F. H. L.

Use of Oxygen in the Ignition of Precipitates. **H. Zöpfchen.** (*Chem. Zeit.*, 1901, xxv., 1008.)—Whenever a filter bears a precipitate which causes the carbon of the paper to be slow in burning off it may with advantage be placed, while still damp, in a porcelain, or preferably platinum Rose crucible, and ignited in a gentle current of oxygen. The process saves much time, and avoids all danger of any reducing action. F. H. L.

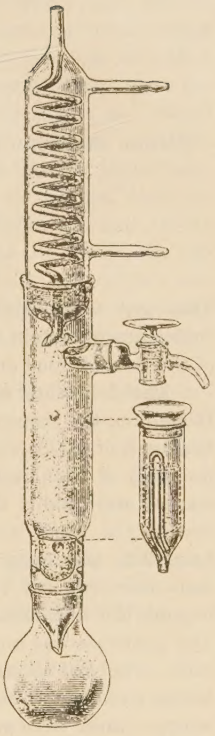
APPARATUS.

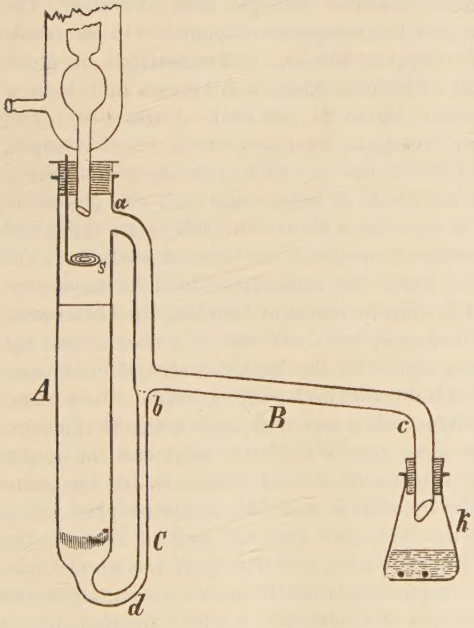
A Heat Concentrator for Ignitions and Concentrations. **C. Jung.** (*Chem. Zeit.*, 1901, xxv., 1113.)—The construction and object of this device are sufficiently explained by the diagram. It introduces a current of hot air and combustion products into a crucible, and vastly hastens ignition, etc. By modifying somewhat the shape of the deflector, returning upwards the central part so as to form a short chimney, it can be placed over an evaporating basin, and renders the whole a kind of surface-evaporator as well. The apparatus is made by W. J. Rohrbeck's Nachfolger of Vienna.



F. H. L.

Combined Apparatus for Extraction and Recovery of the Solvent. A. Chatelan. (*Chem. Zeit.*, 1901, xxv., 612.)—As shown by the accompanying sketch, this apparatus is constructed with a ground joint between the extraction vessel and the condenser, the lower opening of the latter being bent to one side. When extraction is finished, the condenser is rotated through an angle of 180°, so that the volatile liquid falling from the worm drops into the opening at the inner end of the lateral stop-cock. This cock being opened, the solvent can be collected in any suitable receiver. During extraction the cock is closed. The material to be extracted is weighed in the small separate bottle, which is provided with a siphon, and also with a flat stopper to serve as a base when it is on the balance. F. H. L.





An Apparatus for Extracting Liquids with Chloroform. F. Pregl. (*Zeit. anal. Chem.*, 1901, xl., 785-787.)—The construction of the apparatus devised by the author for the extraction of alkaloids by Kippenberger's method is clearly shown in the accompanying figure. The drops of chloroform falling from the condenser are broken up into smaller drops by the wire spiral *S*, thus facilitating the extraction.

C. A. M.

MILK-BLENDED BUTTER.

KING'S BENCH DIVISION.

(Before the Lord Chief Justice of England, Mr. Justice Darling, and Mr. Justice Channell.)

PEARKS, GUNSTON, AND TEE (LIMITED) *v.* HOUGHTON.

From the "Times" of February 27, 1902.

This was a case stated by the Justices of Richmond, Surrey, raising a question as to the sufficiency of certain notices in reference to milk-blended butter within the meaning of Section 8 of the Sale of Food and Drugs Act, 1875.

The following facts appeared from the case stated: An information was preferred by Houghton, the respondent, against Pearks, Gunston, and Tee (Limited), the appellants, under Section 6 of the Sale of Food and Drugs Act, 1875, charging them with unlawfully selling to the prejudice of the purchaser half a pound of shilling butter which was not of the nature, substance, and quality demanded, having had water added to it to the extent of 7·8 per cent. beyond the usual limit of 16 per cent. natural to butter. The appellants were convicted and fined £20 and costs. The appellants were grocers and provision merchants at Richmond and elsewhere, and the respondent was an inspector under the Act. The respondent sent a boy into the appellants' shop to buy half a pound of shilling butter for the purpose of analysis. The purchase was made and the respondent entered the shop, and the butter was handed to him. The Public Analyst certified that the butter contained 23·8 per cent. of water. The butter was handed out by the shop assistant, wrapped in two pieces of paper. On the inside wrapper there were printed in large type the words "Pearks' Butter," and underneath, in much smaller type, a statement that the butter was blended with pure English full-cream milk by new and improved machinery, whereby it retained about 20 to 24 per cent. of moisture. The respondent's attention was not called by the shop assistant to what was on the inside wrapper, and the respondent, though he saw it, did not read it. It was proved by the appellants that it was a common practice in the trade to blend different kinds of butter, and that the process of blending adopted by the appellants was to put the butter into a churn with full-cream milk, and this was re-churned. Any excess of water in the butter in question was derived solely from the milk so added during this process of blending. No water was separately added to the butter. It was stated in evidence on behalf of the appellants that the object of blending the butter with milk is to give it uniform colour and flavour and freshness, but there was no evidence that the milk was added because it was required for the preparation of the butter as an article of commerce fit for carriage or consumption. The price of full-cream milk is ¾d. a pound. In a frame on the wall behind the butter-counter in the appellants' shop was a printed notice to the same effect as that on the wrapper. The butter-counter faces the front of the shop, and the notice was visible to anyone going into the shop; but the respondent did not observe it, and his attention was not called to it. It was contended for the appellants that the inside wrapper was a label within Section 8 of the Sale of Food and Drugs Act, 1875, that the printed notice in the shop was sufficient notice to the respondent apart from the label, and that there was no evidence of fraud. It was further contended that, as the wrapper and printed notice accurately stated the nature and composition of the article sold, there was no evidence of a sale to the prejudice of the purchaser within Section 6 of the Act. It was contended for the respondent that the sale was complete before he entered the shop, and that notice was then too late, and that, as the butter was delivered in a piece of plain, opaque paper, which prevented the printing on the

inside wrapper being visible until removed, and as the respondent did not read it, and did not purposely abstain from reading it, and his attention was not called to it, it was not notice within Section 8, and that as he did not observe the notice on the wall, and his attention was not called to it, it was no notice to him at all. The justices were of opinion that the printed matter on the inside wrapper was misleading, and therefore not sufficient notice within Section 8, as it stated the result of the blending to be to "retain" in the butter about 20 to 24 per cent. of moisture, whereas the true result was to "add" 7·8 per cent. of water to the extreme limit of 16 per cent. natural to butter. They also held that for the same reasons the notice on the wall was not sufficient, that the notice on the inside wrapper was not sufficient within Section 8, that, in the circumstances, the notice on the wall was no notice to the respondent, that the notice was too late, and that the excess of water was added fraudulently to increase the bulk and weight, and notice was therefore no protection. The questions for the court were: (1) whether the inside wrapper was a sufficient notice by label within Section 8; (2) whether there was sufficient notice otherwise than by label; and (3) whether there was any evidence of fraud.

Mr. Asquith, K.C., Mr. Avory, K.C., and Mr. H. D. Bonsey were for the appellants; Mr. Alex. Glen was for the respondent.

Mr. Asquith, for the appellants, cited "Sandys *v.* Small" (3 Q.B.D., 449), "Jones *v.* Jones" (58 J.P., 653), "Spiers and Pond *v.* Bennett" (1896, 2 Q.B., 65), and "Pope *v.* Tearle" (L.R. 9, C.P., 499). He contended that the notices were sufficient, and that there was no evidence of fraud.

Mr. Glen, for the respondent, submitted that there was evidence to support the finding of fraud, as the onus in this respect was on the appellants under Section 24 of the Act. He cited "Pearks *v.* Knight" (1901, 2 K.B., 825), "Liddiard *v.* Reece" (44 J.P., 233), and "Horder *v.* Meddings" (44 J.P., 234).

The Lord Chief Justice, in giving judgment, said that the case was not stated in a satisfactory way; and he did not express any opinion that, if the court was satisfied that this article had been sold as butter and nothing else, there would not be evidence of an offence under Section 6. That was practically decided in "Pearks *v.* Knight," and his Lordship would have come to the same conclusion. But that was not the way in which they had to regard this case. The butter was asked for as half a pound of 1s. butter. If it should turn out that there were various classes of butter in the establishment, and that this was merely butter at a certain price, a different effect had to be given to the notice put up in the shop. He understood the facts to be that the appellants were selling one sort of butter only, by which he meant blended butter. That was the conclusion he drew from the facts, and he did not intend anything he said to apply to a man who sold different kinds of butter and put up this notice. Speaking for himself only, he was not satisfied that if the case had been made out of an offence under Section 6 the inner label would have protected the appellants, but he did not think that was the essential point. If they were dealing with Section 8 only they would have to consider to a greater extent the words of the section "intended fraudulently to increase its bulk," and say whether the person was supplied with a notice at the time. For himself, he had a doubt whether on the delivery of such an article a notice on the inside wrapper was sufficient. "Jones *v.* Jones" was not a sufficient authority for the appellants on that point. He did not think that any one buying a pound of butter and having it delivered to him would be held to have general notice that there was another label inside. If a man had asked for butter and got the article now in question, and the only defence was the label, he was not prepared at present to say that the appellants' contention was right. He understood the magistrates to find that the ordinary purchaser would see the notice on the wall, and he did not think this was qualified by the statement that the respondent did not see it. They were bound by the case of "Sandys *v.* Small" and "Spiers and Pond *v.* Bennett," which decided that if the purchaser was told, not necessarily orally, at the time of the purchase that the article sold was a blended article, it could not then be said that it was sold

to the prejudice of the purchaser. No question of Section 24 and the onus of proof arose here. The prosecution had to prove that there was a sale to the prejudice of the purchaser. It was good law and good sense that, when a man was told that what he was buying was butter blended with something else, he was not entitled to say that he was prejudiced because he thought he was buying butter not blended. To a person who understood the chemistry of butter it would be wrong to say that it "retained" 20 to 23 per cent. of water, unless there was a discussion as to what it was mixed with. To the ordinary purchaser the notice contained a statement that the butter was mixed with milk and had in it 23 per cent. of moisture. He did not think that any supposed misleading statement in that notice would do away with the broad statement that the butter sold in that establishment was in accordance with the notice. He thought there was no evidence of an offence on the ground of prejudice to the purchaser. The magistrates had found that the excess was added fraudulently to increase the bulk and weight. But, if there was no offence under Section 6, the question of supposed fraudulent addition did not arise. He could see no evidence of fraud, but he would hesitate to overrule that class of finding if it was essential to listen to it in order to arrive at justice in a case of the kind.

The other learned JUDGES concurred, and the appeal was accordingly allowed and the conviction quashed, with costs.

REVIEW.

LABORATORY COMPANION TO FATS AND OILS INDUSTRIES. By Dr. J. LEWKOWITSCH, M.A., F.I.C. London: Macmillan and Co. Price 6s. net.

The book, which consists principally of tables, is essentially a companion to the author's "Chemical Analysis of Oils, Fats, and Waxes." Part I. contains, in addition to a number of tables, some pages of matter explanatory of the author's system of fats and oils, which he considers to have been considerably strengthened since the appearance of his "Chemical Analysis." Part II. consists of a number of tables giving the constants of fats, oils, waxes, and the commercial products derived from them, and this will no doubt be found the most useful portion of the book. Part III. contains tables to which frequent reference has to be made in the course of the analysis of fats, oils, etc. The work will be extremely serviceable to those engaged in this special branch of analysis. W. J. S.

BRITISH COLUMBIA INSTITUTE OF ASSAYERS.

AT a provisional meeting held at Nelson, British Columbia, on November 8, 1901, it was unanimously resolved that an Association of Assayers should be formed with the above title, having the following objects in view: The study and promotion of assaying and metallurgical chemistry, and the advancement of the profession generally. No candidate is to be admitted as a member of the Institute unless he is certificated under the Bureau of Mines Amendment Act of 1899. The following officers have been elected for the session 1901-02: President—H. Carmichael, Government Assayer, Victoria. Vice-President—A. McKillop, Nelson. Secretary-Treasurer—J. Cuthbert Welch, Trail. Ordinary Members of Council—A. A. Cole, Rossland; H. Harris, Nelson; Thos. Kiddie, Van Anda; Wm. F. Robertson, Prov. Mineralogist, Victoria.